OTTAWA HULL KIA OCO

BEST AVAILABLE COPY

(11) (C) 1,331,144

(21) 602,929

(22) 1989/06/15

(45) 1994/08/02

(52) 167-304

MARQUES

DROITS (51) INTL.CL.

(51) INTL.CL. A61K-007/075

D'AUTEUR
DESSINS
INDUSTRIELS

(19) (CA) CANADIAN PATENT (12)

Topographies de circuits intégrés

(54) Shampoo Composition

PATENTS
....
TRADB-MARKS

(72) Murray, Andrew M. , U.K.

COPYRIGHT
——
INDUSTRIAL
DESIGN

(73) Unilever PLC, U.K.

INTEGRATED CIRCUIT TOPOGRAPHY

(30) (GB) U.K. 8814296.3 1988/06/16

(57) 16 Claims

1+1

Consommation et
Affaires commerciales Canad

Consumer and Corporate Affairs Canada

3469

Canadä

_ 1 _

J.3080

SHAMPOO COMPOSITION

FIELD OF THE INVENTION

This invention relates to shampoo compositions and more particularly to anti-dandruff shampoos containing an antimicrobial agent which is soluble in an aqueous solution of anionic detergent.

10 Certain 1-hydroxy-2-pyridone derivatives can be used as anti-dandruff agents. These agents have conventionally been employed in shampoo formulations containing anionic detergents such as lauryl ether sulphates or lauryl sulphates. Substances such as anti-dandruff agents must be retained on washed surfaces in order to produce their intended effect. The present invention is concerned with enhancing the retention of such anti-dandruff agents onto hair and scalp after the shampooing process while preserving the optimum foaming properties of the composition.

1331144

GB 1 440 975 (Hoechst) discloses a composition containing 1-hydroxy-2-pyridone or derivatives thereof and surfactants, including sulphosuccinic acid half- and di-esters. There is no indication that any particular anionic surfactant gives foam properties that are superior to other such surfactants which may be included in the composition.

10

15

30

EP 117 135 (Johnson & Johnson) describes a composition which includes anionic surfactant (e.g. monoalkylsulphosuccinate) and an anti-bacterial agent (e.g. 1-hydroxy-2-pyridone or its derivatives). The composition contains also a cationic polymer to enhance retention of the anti-bacterial agent. There is no suggestion that the anionic surfactant would, in the absence of the polymer, enhance retention in this manner.

20 EP 23 676 (REWO) reports a synergistic effect between an anti-dandruff agent and a sulphosuccinate half ester. However, the anti-dandruff agent employed is a pyridinethione derivative.

25 BACKGROUND TO INVENTION

The Applicant has now found that a shampoo having optimum foaming properties, and giving enhanced retention of antimicrobial agent, can be formulated by employing an anti-microbial agent which is soluble in the surfactant together with particular dialkylsulphosuccinate surfactants.

In the case of conventional detergent actives, a reduction in the level of surfactant leads to improved retention of the anti-microbial agent on the skin.

However, there is a corresponding reduction in the foam produced when shampooing. With most detergent actives, high levels of surfactant are needed to provide adequate foam.

5

10

15

20

Applicants have found surprisingly that by formulating a shampoo to combine a low solubility anti-microbial agent with dialkylsulphosuccinate, there results better retention of the same level of the anti-antimicrobial agent than would occur with an equivalent amount of normal high foaming active such as sodium lauryl ether sulphate 2EO. Alternatively, equivalent retention of the anti-microbial agent may be achieved from a composition containing dialkylsulphosuccinates as from a composition containing conventional high foaming actives with a higher level of the same anti-microbial agent. This means that the amount of expensive anti-microbial agent needed is minimised. In addition, the shampoo foams better than would a conventional surfactant system with equivalent retention.

DEFINITION OF THE INVENTION

The invention provides an aqueous shampoo composition comprising, in addition to water:

- (a) dialkylsulphosuccinate, and
- (b) a soluble anti-microbial agent chosen from

 1-hydroxy-2-pyridone,

 1-chlorophenoxy,1-imidazolyl-2-butanone or

 derivatives thereof,
- wherein the dialkylsulphosuccinate is a sodium or ammonium dialkylsulphosuccinate with alkyl chain lengths of from C_6 to C_9 or combinations thereof.

DISCLOSURE OF THE INVENTION

(a) Dialkylsulphosuccinate

The composition according to the invention comprises dialkylsulphosuccinate which is an anionic surfactant. The dialkylsulphosuccinate is present as the sodium or ammonium salt and has alkyl chain lengths of from C₆ to C₉ or combinations thereof. The alkyl chains may be the same or different, and suitably the average number of carbon atoms in the alkyl chains is from 6.5 to 8.5 carbon atoms per molecule of dialkylsulphosuccinate.

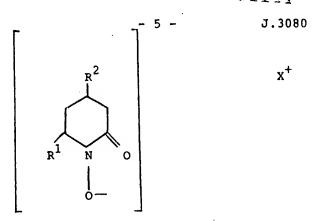
Preferred alkyl chain lengths are combinations of C_6 and C_8 , and the dialkylsulphosuccinate which is especially preferred has alkyl chain lengths $C_6:C_8$ in the ratio 40:60.

The dialkylsulphosuccinate is present in the composition in an amount from 1 to 40% by weight, and preferably from 5 to 20% by weight.

(b) Anti-microbial agent

The composition according to the invention also comprises a soluble anti-microbial agent chosen from 1-hydroxy-2-pyridone, 1-chlorophenoxy, 1-imidazolyl-2-butanone or derivatives thereof. The anti-microbbial agents are soluble in the surfactant system of the shampoo composition.

The preferred 1-hydroxy-2-pyridone derivatives are those of the formula



10 where R¹ is:

5

15

20

25

30

35

1-17C alkyl, 2-17C alkenyl, 5-8C cycloalkyl, 7-9C bicycloalkyl, a cycloalkyl-alkyl having 1-4C alkyl, where the cycloalkyl residue may be substituted by a 1-4C alkyl, aryl, aralkyl having 1-4C alkyl, arylalkenyl having 2-4C alkenyl, aryloxyalkyl or aryl mercaptoalkyl having 1-4C alkyl, benzhydryl, phenyl sulphonylalkyl having 1-4C alkyl, furyl or furyl-alkenyl having 2-4C alkenyl, where the aryl residue may be substituted with a 1-4C alkyl, 1-4C alkoxy, NO₂, CN, or halogen;

and R, is:

H or 1-4C alkyl, alkenyl or alkynyl having each 2-4C, halogen, phenyl or a benzyl group;

and X is an organic amine.

The preferred 1-hydroxy-2-pyridone derivative is the compound known as piroctone olamine, whose chemical name is 1-hydroxy-4-methyl-6-(2,4,4-trimethylpentyl)-2-pyridone monoethanolamine salt, and which is sold under the trade name OCTOPIROX by Hoechst AG.

* denotes trade mark

The anti-microbial agent may also be a 1-chlorophenoxy, 1-imidazolyl-2-butanone, or a derivative thereof. The preferred derivative is 1-(4-chlorophenoxy)-1-(1-imidazolyl)-3,3-dimethyl-2-butanone, available commercially from Bayer under the trade name of Climbazole.

The soluble anti-microbial agent is present in the composition in an amount from 0.01 to 10% by weight, and preferably from 0.1 to 2% by weight.

(c) Optional ingredients

The composition of the invention may also comprise

other ingredients. Optionally, an additional active may
be included so as to improve the retention of the
anti-microbial agent on the skin. Examples of such
additional actives are alkyl sulphate, alkyl ether
sulphate, betaine, amine oxide or mixtures thereof. The
additional surfactant is suitably present in the
composition in an amount of from 1 to 5% by weight.

A hydrotrope, or viscosity and solubility control system, may optionally be included in the composition. An example of a hydrotrope suitable for this purpose is urea. The composition may suitably comprise from 0 to 30% by weight of hydrotrope.

The shampoo composition of the invention may also
include minor amounts of other ingredients which are
commonly employed in shampoos. Examples of such
ingredients are foam boosters, viscosity-adjusting agents,
opacifiers, pearlescers, perfumes, dyes, colouring agents,
conditioning agents, preservatives, thickeners, proteins,
polymers and buffering agents.

denotes trade mark

(d) Water

The composition according to the invention also comprises water in an amount 98.99% by weight or less, forming the balance of the composition.

THE ASSESSMENT METHOD

In salon tests, the assessment method used was the half head shampooing test. Each test involved 40 10 panellists. The hair on each panellist's head was made damp with water, then a 3ml dose of one composition to be tested was applied to one half of the head. Simultaneously, a 3ml dose of a second composition was applied to the other half of the head. The compositions 15 were massaged into the head, without mixing the two, for 1 minute. The hair was rinsed, then dosed with the two shampoo compositions and massaged as before. The two halves of the head were scored by an observer using a Magnitude Estimation method. By this means, either the 20 amount of foam or the anti-dandruff efficacy could be assessed.

OTHER ASPECTS OF THE INVENTION

25

5

Although the invention has so far been described with reference to enhanced retention of an anti-microbial agent from a surfactant system which is a shampoo composition, the principle of retention of a low solubility component is more widely applicable. For example, the retention of sunscreens, perfume or actives other than anti-microbial agents from shampoo or other hair care compositions could be enhanced in this way, in the presence of dialkylsulphosuccinates as defined herein.

35

J.3080

PROCESS

The shampoo composition of the invention is formulated by mixing together the required ingredients in the amounts specified.

EXAMPLES

The examples below are illustrations of shampoo compositions of this invention. In examples 1, 2, 3, 5 and 6, comparative shampoo formulations are included to illustrate the superior efficacy of the compositions of the invention. The abbreviations used signify the following compounds:

15

5

DIAS - dialkylsulphosuccinates of chain lengths $C_6:C_8$ in the ratio 40:60

SLES - sodium lauryl ether sulphate (2EO)

CDE - coconut diethanolamide (foam booster)

20

Example 1

	Ingredient	8 1	N/W
		<u>A</u>	<u>B</u>
25 [.]			
	DIAS	0	11.0
	SLES	16.0	0
	Octopirox	0.75	0.3
	Water	to 100%	to 100%

30

35

In vitro studies were conducted which involved treating samples of animal skin, as a model for the human scalp, with composition A or B. It was shown that the Octopirox retention of B was equivalent to that of A. This demonstrates the superior efficacy of Octopirox with

dialkylsulphosuccinates by comparison with compositions containing conventional surfactants with higher levels of Octopirox.

5 Example 2

	Ingredient	<u>8</u>	8 W/W	
		<u>A</u>	<u>B</u>	
10	DIAS	0	9.0	
	SLES	12.0	0	
	CDE	2.0	0	
	Octopirox	0.3	0.3	
	Water	to 100%	to 100%	

15

The surfactant system B was shown in vitro to produce an equivalent foam performance to that of A. Thus, a composition containing dialkylsulphosuccinates gives equivalent foam performance to that containing a higher level of conventional surfactant.

Example 3

25	Ingredient	<u>8 w</u>	<u>% w/w</u>		
		<u>A</u>	B		
	DIAS	0	11.0		
	SLES	16.0	0 .		
30	Octopirox	various	various		
	Water	to 100%	to 100%		

The results of $\underline{\text{in vitro}}$ tests of compositions A and B containing different levels of Octopirox on samples of

animal skin are shown in Figure 1. The amount of Octopirox in compositions A and B is plotted against the level of Octopirox retained per cm² of skin, and it can be seen that, for each level of Octopirox, the retention of Octopirox from B is approximately twice that from A.

Example 4

	Ingredient	. 8 1	W/W
10		<u>A</u>	<u>B</u>
	DIAS	11.0	11.0
	SLES	0	1.0
	Octopirox	0.4	0.4
15	Water	to 100%	to 100%

Comparative testing in vitro showed that the amount of Octopirox retained per cm² of skin was 1.8µg for A and 2.3µg for B. Thus, the retention of Octopirox from B was superior to that from A, and it can be concluded that retention of DIAS is further enhanced by the addition of SLES to a DIAS base.

25 Example 5

	Ingredient	<u>% w/w</u>		
		<u>A</u>	<u>B</u>	
30	DIAS	0	11.0	
	SLES	16.0	1.0	
	CDE	3.0	0	
	Octopirox	0.75	0.4	
	Water	to 100%	to 100%	

-11-

Salon tests (half head shampooing tests) showed that the foam produced by B was equivalent to that produced by A.

Example 6

	Ingredient	8 W	8 w/w	
•		<u>A</u>	B	
10	DIAS	0	11.0	
	SLES (2EO)	16.0	1.0	
	CDE	3.0	0	
	Octopirox	0.75	0.4	
	Water	to 100%	to 100%	

15

In clinical tests (half head shampooing tests), composition B had an anti-dandruff efficacy equivalent to that of composition A.

20

Example 7

	Ingredient	<u>% w/w</u>
25	DIAS	13.0
	SLES	1.0
	Octopirox	1.0
	Water	to 100%

The above composition in accordance with the invention was compared with a commercially-available shampoo containing ammonium lauryl ether sulphate and ammonium lauryl sulphate as surfactant system and 1% zinc pyridinethione as anti-dandruff agent.

In clinical tests (half-head shampooing tests) it was shown that the above composition had an anti-dandruff efficacy significantly better than that of the commercially-available shampoo.

5

The invention is further illustrated by the following Examples.

Example 8

10

	Ingredient	8 w/w
	DIAS	11.0
	(C ₆ :C ₈ in a ratio of 40:60)	
15	Opacifier	5.0
	Urea	1.0
	Octopirox	0.4
	Thickener	2.0
	Perfumes, preservatives	q.s
20	Water	to 100%

Example 9

	Ingredient		8 w/w
	DIAS		13.0
	$(C_6:C_8)$ in a ratio of $40:60$)		
30	Lauryl betaine (30% active)		7.0
	Urea		10.0
	Octopirox		1.0
	Thickener		2.0
	Perfumes, preservatives		q.s
35	Water	to	100%

Example 10

			% wt
5	Dialkyl suphosuccinate		12.0
	(di-C _B)		
	Octopirox		0.4
	Urea		10.0
	Thickener		2.0
10	Perfumes/preservatives		q.s.
	Water	to	100

Example 11

15

		8 wt
	Dialkylsulphosuccinate	12.0
	(C ₆ :C ₈ in a ratio of 40:60)	
20	Urea	10.0
	Climbazole	0.1
	Thickener	3.0
	Perfume/preservative	q.s.
	Water to	100

25

Example 12

		8 wt
30	Dialkylsulphosuccinate	12.0
	(C ₆ :C ₈ in a ratio of 40:60)	
	Urea	5.0
	Opacifier	5.0
	Climbazole	0.6
35	Perfume/preservative	q.s.
	Water to	100

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

- 1. An aqueous shampoo composition comprising, in addition to water:
 - (a) dialkylsulphosuccinate, and
- B (b) a soluble anti-microbial agent chosen from 1-hydroxy-2-pyridone, 1-chlorophenoxy, 1-imidazolyl-2-butanone or derivatives thereof,

wherein the dialkylsulphosuccinate is a sodium or ammonium dialkylsulphosuccinate with alkyl chain lengths of from ${\rm C}_6$ to ${\rm C}_{\rm Q}$ or combinations thereof.

- 2. An aqueous shampoo composition as claimed in Claim 1 wherein the alkyl chains contain an average of 6.5 to 8.5 carbon atoms per molecule of dialkylsulphosuccinate.
- 20 3. A shampoo composition as claimed in Claims 1 or 2, wherein the dialkylsulphosuccinate has alkyl chain lengths C_6 and C_8 or combinations thereof.
- A shampoo composition as claimed in Claim 1 or 2
 wherein the dialkylsulphosuccinate has alkyl chain lengths
 C₆:C₈ in the ratio 40:60.
- A shampoo composition as claimed in Claim 1 or 2
 wherein the dialkylsulphosuccinate is present in an amount
 from 1 to 40% by weight.
 - 6. A shampoo composition as claimed in Claim 1 or 2 wherein the dialkylsulphosuccinate is present in an amount from 5 to 20% by weight.

35

5.

- 7. A shampoo composition as claimed in Claim 1, wherein the soluble anti-microbial agent is 1-hydroxy-4-methyl-6-(2,4,4-trimethylpentyl)-2-pyridone monoethanolamine salt.
- 8. A shampoo composition as claimed in Claim 1 wherein the soluble anti-microbial agent is 1-(4-chlorophenoxy)-1-(1-imidazoly1)-3,3-dimethyl-2-butanone.
- 10 9. A shampoo composition as claimed in Claim 1, wherein the soluble anti-microbial agent is present in an amount from 0.01 to 10% by weight.

5

20

- 10. A shampoo composition as claimed in Claim 9, wherein 15 the soluble anti-microbial agent is present in an amount from 0.1 to 2% by weight.
 - 11. A shampoo composition as claimed in Claim 1 further comprising from 0.1 to 30% by weight of an additional surfactant.
- 12. A shampoo composition as claimed in Claim 11, wherein the additional surfactant is chosen from an alkyl sulphate, an alkyl ether sulphate, a betaine, or an amine oxide, or mixtures thereof.
 - 13. A shampoo composition as claimed in Claim 11 or Claim 12, wherein the additional surfactant is present in an amount from 1 to 5% by weight.
 - 14. A shampoo composition as claimed in Claim 1, further comprising from 0 to 30% by weight of a hydrotrope.
- 15. A shampoo composition as claimed in Claim 14, wherein35 the hydrotrope is urea.

-16-

16. A shampoo composition as claimed in Claim 14 or Claim 15, wherein the hydrotrope is present in an amount from 1 to 10% by weight.

This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:
BLACK BORDERS
☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
☐ FADED TEXT OR DRAWING
☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
☐ SKEWED/SLANTED IMAGES
COLOR OR BLACK AND WHITE PHOTOGRAPHS
GRAY SCALE DOCUMENTS
☐ LINES OR MARKS ON ORIGINAL DOCUMENT
☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
OTHER:

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.